U. S. NAVAL AIR ENGINEERING CENTER

PHILADELPHIA, PENNSYLVANIA

AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC AML 2454 DATE 16 May 1966

THE CORROSION PROTECTION AFFORDED BY VARIOUS COATING SYSTEMS IN AIRCRAFT FASTENER AREAS

PAN 12-51 UNDER BUWEPS WEPTASK
RRMA 03 003/200 1/R007 03 01

Distribution of this document is unlimited

NOTICE

Reproduction of this document in any form by other than naval activities is not authorized except by special approval of the Secretary of the Navy or the Chief of Naval Operations as appropriate.

The following Espionage notice can be disregarded unless this document is plainly marked CONFIDENTIAL or SECRET.

This document contains information affecting the national defense of the United States within the meaning of the Espionage Laws, Title 18, U.S.C., Sections 793 and 794. The transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

NAVAL AIR ENGINEERING CENTER
PHILADELPHIA, PENNSYLVANIA 19112

AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC AML 2454 DATE 16 May 1966

THE CORROSION PROTECTION AFFORDED BY VARIOUS COATING SYSTEMS IN AIRCRAFT FASTENER AREAS

PAN 12-51 UNDER BUWEPS WEPTASK
RRMA 03 003/200 1/R007 03 01

Distribution of this document is unlimited

PREPARED BY: Jack Ohr
JACK OHR
PROJECT ENGINEER

APPROVED BY: A. I. Falkowitz
A. I. FALKOWITZ, HEAD
ORGANIC PROTECTIVE
COATINGS BRANCH

G. K. Holmes
G. K. HOLMES, SUPERINTENDENT
CHEMICAL ENGINEERING
DIVISION

TABLE OF CONTENTS

LIST OF TABLES AND PLATES

ABSTRACT

A stress-cycling test (at -103°F and room temperature) designed to simulate the spectrum of stresses which could occur during 500 aircraft flying hours (roughly 1 PAR interval), indicates that current specification and experimental aircraft coatings cannot meet these conditions without cracking around fastener heads.

The coatings used were the MIL-C-22750 epoxy, the MIL-L-81352(WEP) all-acrylic, and two polyurethanes.

The implication of this test is that, at the present state of the coatings art, paint films of the non-rubbery type cannot be expected to provide adequate corrosion protection by themselves in the fastener area-a persistent trouble area on naval aircraft.

I. INTRODUCTION

A. References (a) and (b) established this problem assignment to develop improved corrosion resistant primers for protection of aircraft, with specific emphasis on the protection of aluminum countersinks fitted with cadmium plated steel fasteners.

B. Reference (c) was a report which indicated that aircraft fastener areas (cadmium plated steel fasteners in aluminum countersinks) could be effectively protected against corrosion by treatment with a MIL-S-8802 sealant, Products Research Company's PR1422 (B2). The treatment recommended therein was, for maximum protection, PR1422 (B2) in the countersink prior to fastener installation and a PR1422 (B2) overlay after installation. A somewhat less effective technique was the application of the PR1422 (B2) overlay alone for those aircraft where it is impractical to apply the PR 1422 (B2) in the countersinks.

C. The conclusions of the reference (c) investigation were based on stress-cycling tests of MIL-C-22750 epoxy and polyurethane coated aluminum specimens with and without PR1422 (B2) treatment, followed by salt spray tests and examination of the disassembled stripped specimen components.

D. With the exception of one very elastomeric polyurethane system (two were used), all the specimens exhibited noticeable paint cracking around fasteners, including those specimens which had been treated with PR1422 (B2) prior to painting. The explanation for the corrosion protection qualities of the PR1422 (B2) appears to lie in its ability to remain soft and flexible enough to withstand severe stressing, and impermeable enough to withstand moisture and electrolyte penetration even though the paint system is cracked and flaked.

E. The stress tests employed in reference (c), while severe, were not designed to simulate the actual spectrum of stresses encountered by military aircraft, or to correlate with length of service life. It was of interest, therefore, to know if current specification and experimental aircraft coating systems exhibit fastener head cracking after a reasonable length of service life, e.g., one PAR. If, say, the MIL-C-22750 epoxy system cracked after one simulated PAR whereas the MIL-L-81352 all-acrylic or polyurethane did not, then the use of the latter systems is indicated and further protection methods such as PR1422 (B2) may be superfluous. In this connection, it was noted above that the elastomeric polyurethane did not crack during stressing. Even so, corrosion did occur in the fastener area. However, reference (c) has explained that even though the coating was intact, it was nevertheless broken down by exhibiting severe stretching (beyond its elastic limit) around the fastener heads. The stretched coating was probably highly permeable to moisture and electrolytes.

F. Accordingly, the objective of this investigation was to determine which of the current specification and experimental coating systems is most desirable for aircraft use by virtue of its crack resistance around fastener

heads after one simulated PAR tour of duty. Towards this end, the Aeronautical Structures Laboratory of the Naval Air Engineering Center provided the stress-cycling test parameters for simulating the spectrum of stresses encountered by military aircraft during 500 flying hours, which is approximately equivalent to 15 months of duty or 1 PAR. The coating systems tested to these conditions were MIL-C-22750 epoxy, MIL-L-81352 all-acrylic, and two polyurethanes.

II. EXPERIMENTAL PROCEDURES

A. Specimen Preparation:

(1) The Figure 1 specimens were fabricated from unclad 7075 aluminum alloy. The fasteners were cadmium plated steel, AN 509-10R-12.

(2) The unassembled three sections of the specimens were treated with Iridite 14-2 (including countersinks). The faying surfaces were then shop-primed with two coats of MIL-P-23377 epoxy primer, dried overnight, and the specimens assembled.

(3) Just prior to fastener installation, the countersinks were treated with primers, sealant, or left untreated. When treated, the primers or sealant were wet-applied by stirring rod and the fasteners immediately installed at a torque of 25 in.-lb. The excess primer or sealant was wiped from around the fastener heads with a clean cloth. (These test areas will be utilized in future corrosion work not included in this report.)

(4) The installed fastener heads were treated for 1½ minutes with MIL-C-5410 (1 x 1 water) and then flushed off with water. The heads were then brush treated for about 5 minutes with Iridite 14-2 containing ARP #2 detergent for better wetting.

(5) At this stage, a band of Products Research Company's PR1422 (B2) sealant was applied over the fastener head areas of some of the specimens. The band was about 6-8 mil thick and ½ inch wide for each fastener row and was applied across the entire width of the specimen.

(6) A 1/32 inch thick 1½ inch cross was made at the top and bottom of each specimen with masking tape so that a bare test area could be had after painting. (These test areas will be utilized in future corrosion work not included in this report).

(7) The specimens were then primed and topcoated with the epoxy, acrylic, or polyurethane systems according to the coating schedules of Tables 1 and 2. The taped crosses were now removed exposing an unpainted, Iridited test cross. Note that topcoat formulations are listed in Table 3.

(8) After air drying for 1 week, the specimens were exposed on the roof of the Aeronautical Materials Laboratory from late July 1965 to January 1966. The specimens were removed from exposure for a short period (1 week)

so that 1½ x ½ inch unpainted test areas could be machined with a lathe on the top and bottom of the specimens. These areas differed from the above cross area in that the former were machined through the Iridite treatment. (These areas will be utilized in future corrosion work not included in this report).

(9) After exposure, some of the specimens were baked to simulate aerodynamic heating: 90 hours at 260°F, followed by 10 hours at 300°F, followed by 2½ hours at 350°F followed by ½ hour at 400°F. Tables 1 and 2 indicate those specimens which were baked.

(10) A layer of pressure sensitive tape (3M Polyvinyl Fluoride-Y9057) was applied over the fastener heads of one epoxy painted specimen, #218 of Table 1.

B. <u>The Cyclic Loading Test (See Plate 8)</u>:

(1) It has been calculated that if a specimen such as described in Figure 1 were subjected to a pulling force of 11250 lb. along its longitudinal axis and then released to a minimum pull of 1535 lb. so that the specimen is under tension for the complete cycle, then 1000 of such cycles would approximate, on the specimen, the spectrum of stresses which would occur on military aircraft during 500 flight hours (which is roughly equivalent to one PAR cycle or 15 months). It is noted that the 1535 lb. limit represents the minimum stresses to which an aircraft is subjected in flight, corresponding to steady state flying conditions without acceleration or deceleration. The 11250 lb. limit represents a very high stress level corresponding to a 7.33 g maneuver condition which would occur during sharp changes in the aircraft's acceleration or change in direction. Calculation indicates that the actual stress on the narrow width of the specimen when pulled to 11250 lb. is 30,000 lb./in.^{2} (11250 lb. force ÷ cross sectional area, 0.125 x 3 inches). The actual stress on the fastener head at 11250 lb. is difficult to calculate and must be determined empirically for each fastener, but it is estimated that the stress should be about three times the stress on the specimen width, or 90,000 lb./in.^{2}.

(2) Painted stress specimens such as described in Figure 1 and elsewhere in this section were installed vertically in the jaws of a Krouse Fatigue machine which had been programmed to provide the maximum and minimum stresses described above, and also lesser stresses. The ratio of maximum to minimum stress was always 10/1 since this was most practical with the machine. Hence, the max./min. stress for the max 11,250 pull was 11250/1125 instead of the idealized 11250/1535. This decreases the minimum stress level from 4000 lb./in.^{2} to 3000 lb./in.^{2} which is considered a minor error for this test.

(3) Upon installation, some specimens were packed with dry ice by enclosing an insulated metal box about each specimen and filling the box with small dry ice chips. Twenty minutes were permitted for temperature equilibrium prior to the introduction of stresses. After this time, the surface temperature of the specimens was measured by thermocouple to be about -103°F (the dry ice temperature is -109°F.) At this point, the apparatus was adjusted to produce the min. and max. loads and the specimen was cycled. The cycles were read by meter.

Note that not all specimens were stress-cycled at cold temperature conditions-some were cycled at room temperature.

(4) The temperature conditions, number of cycles, stress configuration, and other test details for each specimen are listed in Tables 1 and 2.

III. RESULTS

The below listed results may be placed in clearer perspective by consideration of the stress cycling test parameters which were employed on the Figure 1 specimens to simulate 500 flying hours on 1 PAR: maximum stress, 30,000 lb./in.^{2}; minimum stress, 3000 lb./in.^{2}; number of cycles, 1000; temperature, -103°F.

A. The MIL-C-22750 epoxy, the MIL-L-81352 lacquer, and two polyurethane topcoats exhibited cracking around fastener heads when tested to simulate 1 PAR. (One of the polyurethanes, P114-1, was satisfactory when tested at -60°F as per reference (d), the conical mandrel flexibility test).

B. In many cases, the above coatings exhibited fastener head cracking when tested at conditions notably less severe than the simulated 1 PAR test.

(1) Example: Most specimens, regardless of topcoat, exhibited fastener head cracking when the maximum stress was reduced from 30,000 lb./in.^{2} to 17,500 lb./in.^{2}.

(2) Example: All specimens, regardless of topcoat, exhibited faster head cracking when the simulated 1 PAR test was conducted at room temperature.

C. The coatings in paragraph A. above which were baked to simulate aerodynamic heating offered markedly less resistance to cracking around fastener heads than the same type unbaked coatings.

D. The coatings in paragraph A. which were applied over the PR1422 (B2) sealant strip generally exhibited cracking around fastener heads after the simulated 1 PAR test.

E. The pressure sensitive polyvinyl fluoride tape applied over the fastener heads of one epoxy coated specimen exhibited cracking around fastener heads.

F. High speed motion pictures (400 frames per second) taken of the coated fastener areas during cycling and projected on a screen clearly shows movement between the fastener and surrounding structure. This movement is also apparent in Plates 1-7 which are single frames from the motion pictures and show the paint crack widen and narrow with increased and decreased load during one cycle.

IV. CONCLUSIONS

A. The implication of the stress-cycling test is that it is improbable that any current specification or experimental non-rubbery type coating system, per se, can provide reasonable long-term corrosion protection to aircraft fastener areas.

B. Even the P114-1 polyurethane coating which has unusually good flexibility for a rigid, non-rubbery type suitable for aircraft use (satisfactory to the conical mandrel flexibility test at -60°F) exhibited cracking around fastener heads when evaluated in the simulated 1 PAR test.

V. RECOMMENDATIONS

A. No recommendations are made at this stage of the subject investigation.

REFERENCES

(a) BUWEPS ltr RRMA-52:SK/108 of 3 May 1961

(b) BUWEPS ltr RRMA-52:SK/22 of 23 Feb 1965

(c) Report No. NAEC AML 2346 of 2 Feb 1966

(d) Federal Test Method Standard No. 141, Method 6222, Flexibility, Conical Mandrel Test

ACKNOWLEDGEMENTS

Sincere appreciation is given to Mr. Ralph Vining of the Aeronautical Structures Laboratory for providing the parameters for the stress-cycling test and the design of the specimens, and for his patient instruction in some of the more fundamental aspects of this test; to Mr. Michael Stellabotte of the Metallurgical Division of the Aeronautical Materials Laboratory for his instruction in the use of the Krouse Flexure Apparatus; and to Messrs. John Stewart and Holt Smith for their assistance in operating the K-F apparatus.

CYCLIC LOADING TESTS AT -103°F

Specimen Designation	Coating System(1)	Forces on Specimen-lbs. Max.	Forces on Specimen-lbs. Min.	Stresses on Specimen (lbs./in.^{2}) Max.	Stresses on Specimen (lbs./in.^{2}) Min.	No. of Cycles	Condition of Coating Around Most Fastener Heads	Coating System-Baked Prior to Stressing Yes	Coating System-Baked Prior to Stressing No
14	WPI + P46 + P79 Epoxy	5800	575	15600	1560	1000	Fine cracking		x
	Same specimen	11250	1125	30,000	3000	1000	Slightly more cracking		
6	WPI + P46 + P79 Epoxy	5800	575	15600	1560	1000	Cracking		x
11	WPI + P46 + P79 Epoxy	5800	575	15600	15600	1000	Cracking		x
91	P46 + 95K Acrylic	5800	575	15600	1560	1000	Satisfactory		x
	Same specimen	7000	700	18800	1880	1000	Cracking		
92	P46 + 95K Acrylic	5800	575	15600	1560	1000	Cracking		x
100	P46 + 95K Acrylic	5800	575	15600	1560	1000	Satisfactory		x
	Same specimen	11250	1125	30,000	3000	1	Cracking		
89	P46 + 95K Acrylic	7000	700	18800	1880	1000	Cracking		x
75	P46 + 43MS Polyurethane	5800	575	15600	1560	1000	Cracking		x
80	P46 + 43MS Polyurethane	5800	575	15600	1560	1000	Satisfactory		x
	Same specimen	7000	700	18800	1880	1000	Satisfactory		
	Same specimen	11250	1125	30,000	3000	1000	Cracking		
78	P46 + 43MS Polyurethane	5800	575	15600	1560	1000	Cracking		x
79	P46 + 43MS Polyurethane	5800	575	15600	1560	1000	Satisfactory		x
	Same specimen	7000	700	18800	1880	1000	Satisfactory		
	Same specimen	11250	1125	30,000	3000	1000	Cracking		
81	P46 + 43MS Polyurethane	5800	575	15600	1560	1000	Satisfactory		x
	Same specimen	11250	1125	30,000	3000	1000	Cracking		

TABLE 1

TABLE 1

Designation	Coating System(1)	Forces on Specimen-lbs. Max.	Forces on Specimen-lbs. Min.	Stresses on Specimen ($lbs./in.^2$) Max.	Stresses on Specimen ($lbs./in.^2$) Min.	No. of Cycles	Condition of Coating Around Most Fastener Heads	Coating System-Baked Prior to Stressing Yes	Coating System-Baked Prior to Stressing No
B	P46 + P114-1 Polyurethane	7000	700	18800	1880	1000	Cracking		x
379	P46 + 95K Acrylic	5800	575	15600	1560	1000	Cracking	x	
166	P46 + P43MS Polyurethane	5800	575	15600	1560	1000	Cracking	x	
173	P46 + P43MS Polyurethane	5800	575	15600	1560	1000	Cracking	x	
167	P46 + P43MS Polyurethane	5800	575	15600	1560	1000	Cracking	x	
A	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1000	Cracking	x	
218	P46 + P79 Epoxy + 3M Polyvinyl Fluoride Tape (Y-9057)	11250	1125	30,000	3000	1000	Cracking of tape around fastener heads		x
139	WPI + P46 + P79 over PR1422 ribbon	11250	1125	30,000	3000	1000	Satisfactory-No observable cracking of paint system		x
141	WPI + P46 + P79 over PR1422 ribbon	11250	1125	30,000	3000	1000	Fine cracking		x
142	WPI + P46 + P79 over PR1422 ribbon	11250	1125	30,000	3000	1000	Fine cracking		x

NOTES TO TABLE 1

(1) WPI = Wash primer, MIL-C-8514
P46 = Epoxy primer, MIL-P-23377
P79 = Epoxy topcoat, MIL-C-22750 (Insignia White)
PR1422 = Products Research Company's PR1422, Class B filleting compound. Prepared by mixing 75 parts by weight of Part I to 10 parts by weight of Part II.
P43MS = AML formulated white polyurethane topcoat
P114-1 = AML formulated white polyurethane topcoat
95K = White all-acrylic topcoat, MIL-L-81352(WEP)

TABLE 2

CYCLIC LOADING TESTS AT ROOM TEMPERATURE

Specimen Designation	Coating System	Forces on Specimen-lbs.		Stresses on Specimen (lbs./in.^{2})		No. of Cycles	Condition of Coating Around Most Fastener Heads	Coating System-Baked Prior to Stressing	
		Max.	Min.	Max.	Min.			Yes	No
72	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1000	Cracking		x
73	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1000	Cracking		x
168	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1	Cracking	x	
169	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1	Cracking	x	
99	P46 + 95K Acrylic	11250	1125	30,000	3000	1000	Cracking		x
101	P46 + 95K Acrylic	11250	1125	30,000	3000	1000	Cracking		x
174	P46 + 95K Acrylic	11250	1125	30,000	3000	1	Cracking	x	
175	P46 + 95K Acrylic	11250	1125	30,000	3000	1	Cracking	x	
300	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1	Cracking	x	
301	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1	Cracking	x	
302	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1000	Cracking		x

NOTES: (1) P46 = Epoxy primer, MIL-P-23377; (2) P43MS = AML formulated white polyurethane
(3) P95K = White all-acrylic topcoat; (4) P114-1 = AML formulated white polyurethane topcoat

FORMULATION DATA FOR TOPCOATS

A. Polyurethane, 114-1

Ingredients	Parts by Weight
TiO_2 (R610)	360
Castor Oil #1066, Spencer-Kellog Company	288
Cellosolve Acetate (Urethane Grade)	138
Xylene	138

Grind in pebble mill, then mix 29.8 parts by weight to:

Aliphatic Isocyanate XP1699, Spencer-Kellog Company	343

B. Polyurethane, P43MS

Ingredients	Parts by Weight
Desmophen 650, Mobay Chemical Company	321
TiO_2 (R610)	267
Dri-Film, General Electric Company	0.8
Zinc Naphthenate (8%)	1.2
Cellosolve Acetate (Urethane Grade)	223
Xylene	40
Cyclohexanone	50

Grind in pebble mill, then to 90.3 parts by weight of above, add:

Desmoden N, Mobay Chemical Company	45

C. Acrylic, MIL-L-81352(WEP)

Ingredients	Parts by Weight
TiO_2 (R610)	720
Acryloid A21 (30% solution) Rohm & Haas Co.	1720
Acryloid B44 (40% solution) Rohm & Haas Co.	1284
Santicizer 160, Monsanto Co.	54
Cellosolve Acetate	175

Grind in pebble mill

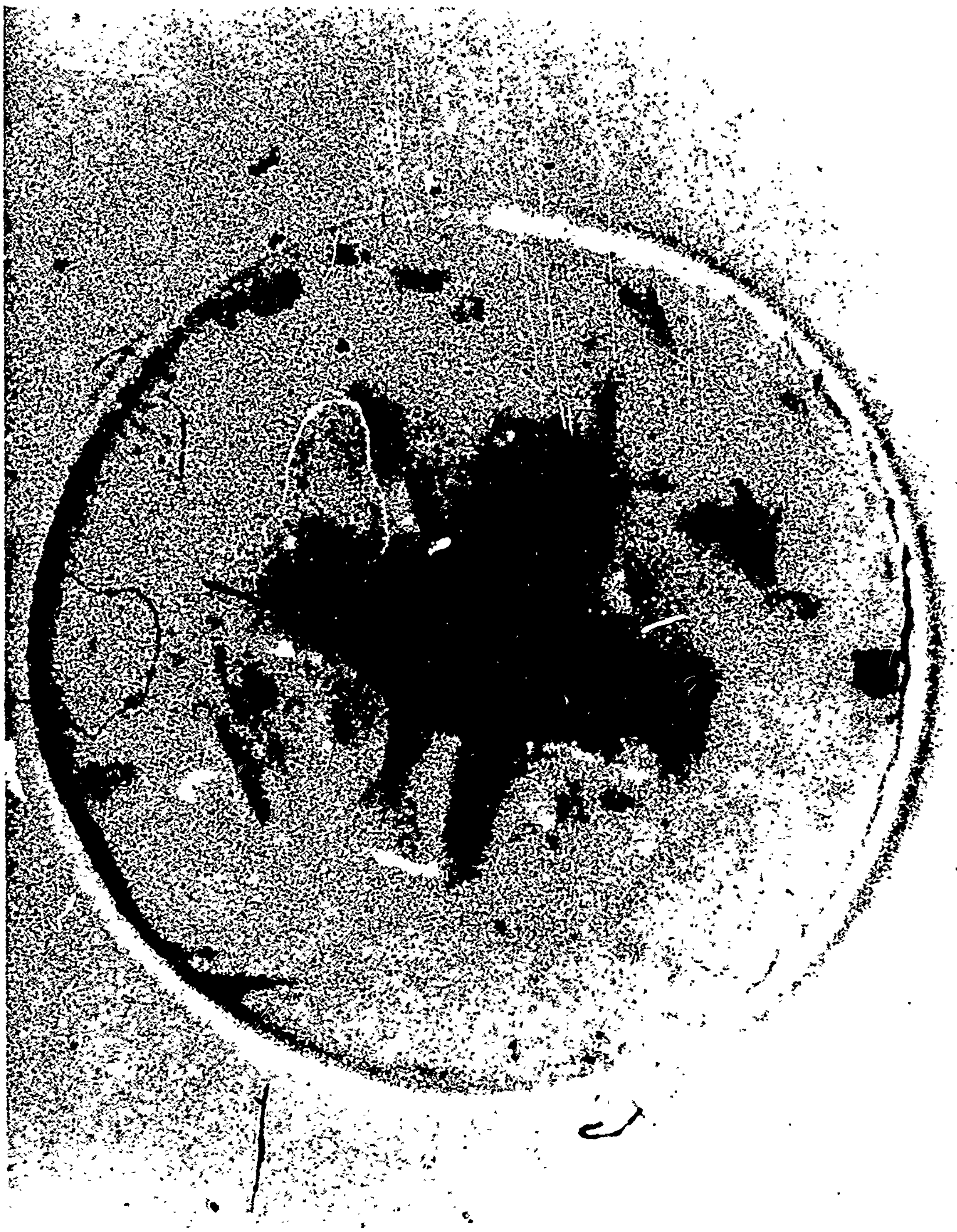
D. Epoxy, MIL-C-22750

See Specification

TABLE 3

1st PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

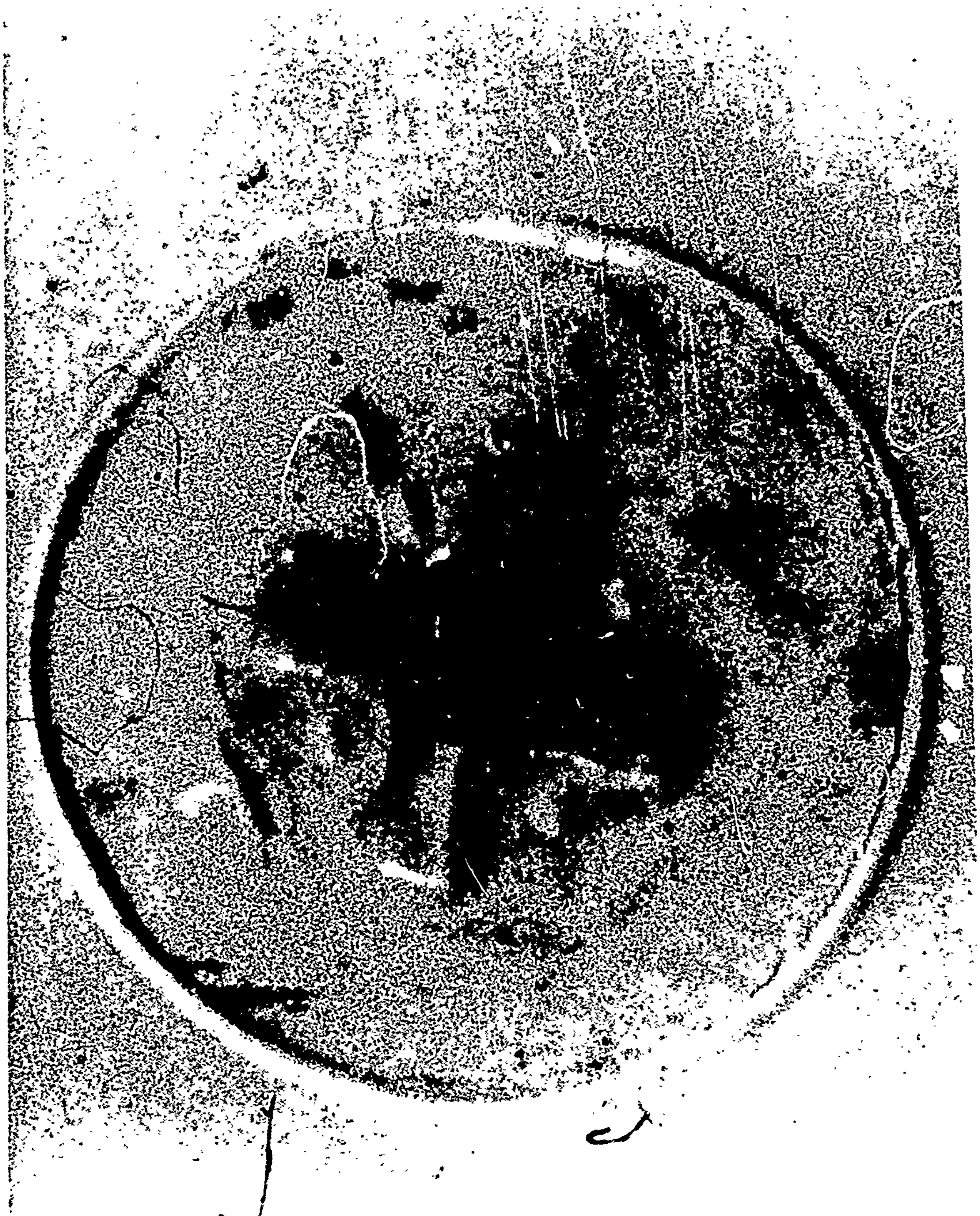
PHOTO NO: CAN-375450(L)-5-66 PLATE 1



2nd PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

PHOTO NO: CAN-375451(L)-5-66

PLATE 2



3rd PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

PHOTO NO: CAN-375452(L)-5-66

PLATE 3

4th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

PHOTO NO: CAN-375453(L)-5-66

PLATE 4

5th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

PHOTO NO: CAN-375454(L)-5-66

PLATE 5

6th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

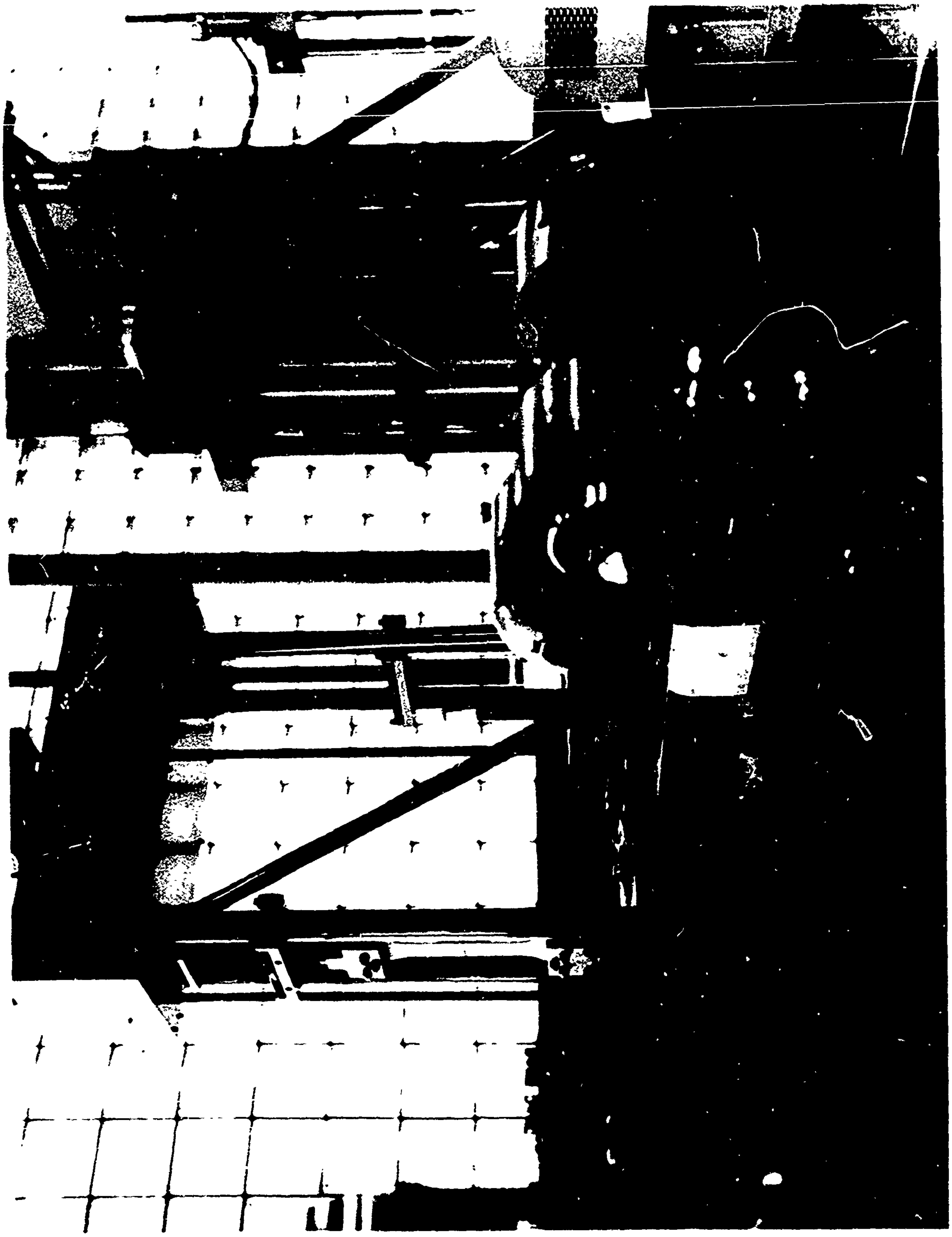
PHOTO NO: CAN-375455(L)-5-66

PLATE 6

7th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST

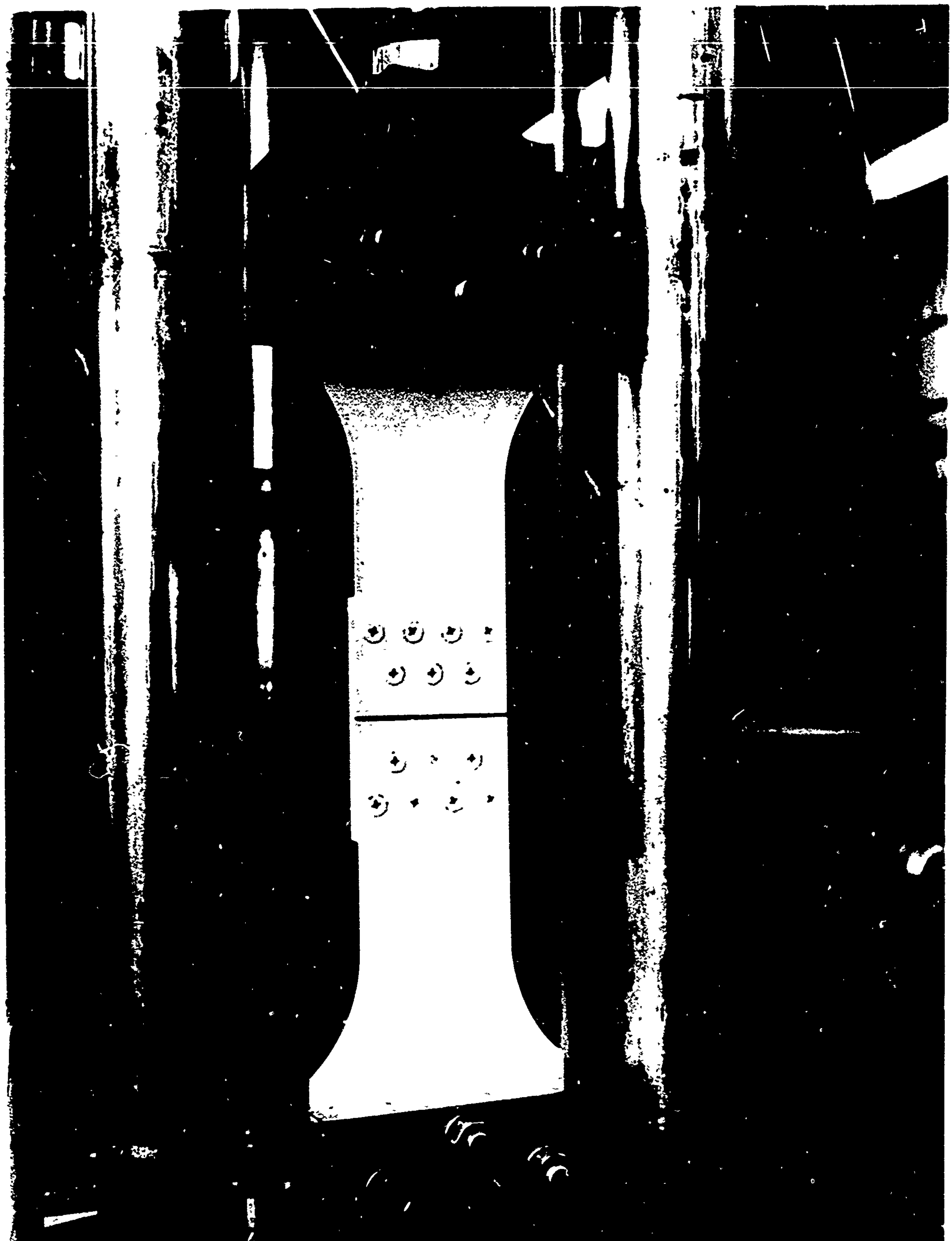
PHOTO NO: CAN-375456(L)-5-66

PLATE 7



KROUSE DIRECT STRESS TESTING MACHINE WITH SPECIMEN MOUNTED

PHOTO NO: CAN-375245(L)-4-66



CLOSE-UP OF MOUNTED SPECIMEN

PHOTO NO: CAN-374854(L)-4-66

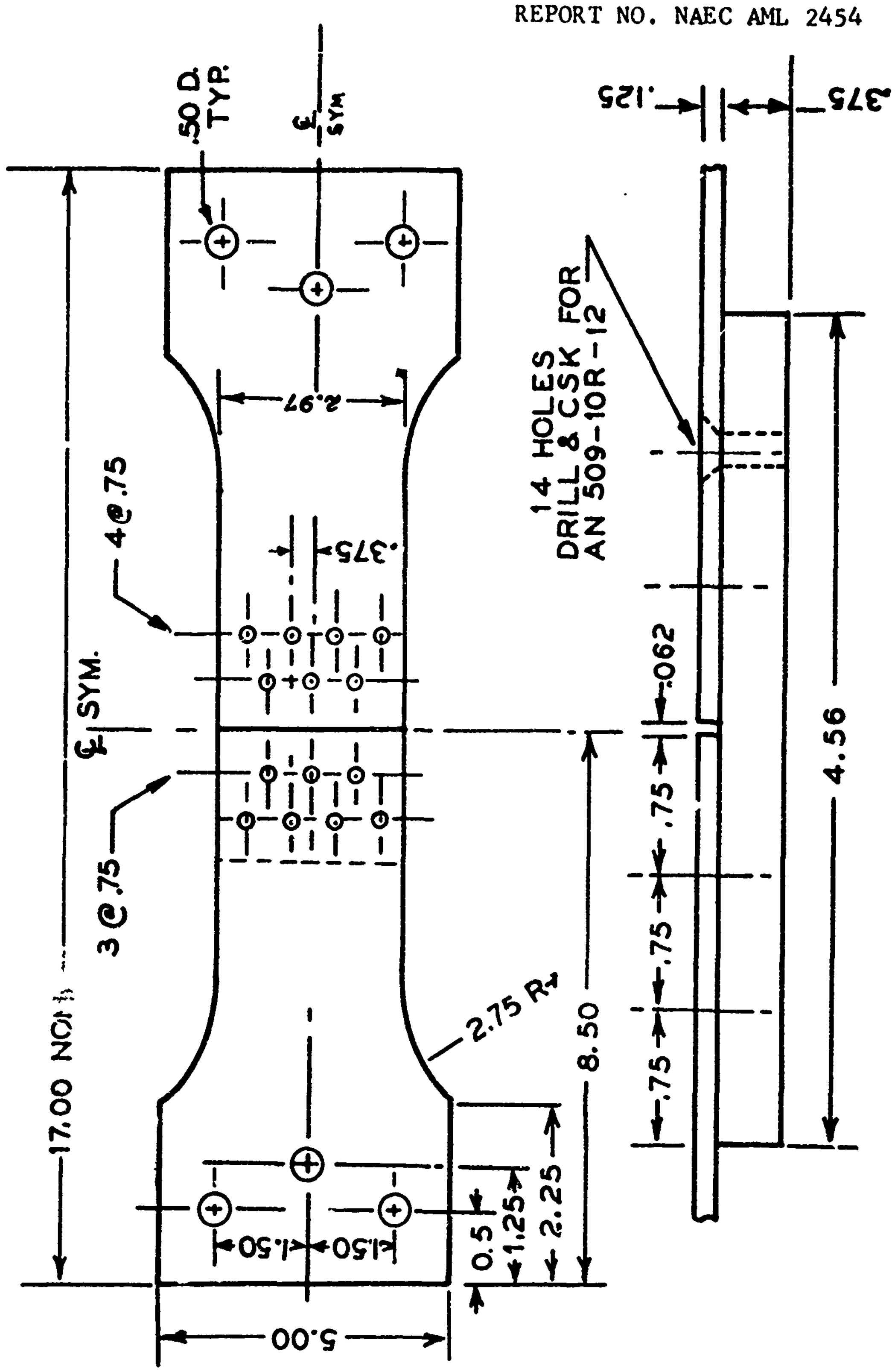


FIGURE 1 - THE STRESS SPECIMEN USED IN THE CYCLIC LOADING APPARATUS